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MEMORANDUM

DATE: 17 November 1998

TO: David Bennett, WAM, U S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
run Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch: K9805449
Site: Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO : 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U S. EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of seven sediment samples, laboratory batch K9805449, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98334012	98334017	98334019	98334020
98334022	98334023	98334024	

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240 1, February 1994), modified to include specific requirements of analytical methods.

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DCN 4000-019-038-AAAK

17 November 1998
Region X





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1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria.

2. Detection Limits—Acceptable

Instrument detection limits met project required quantitation limits.

3 Initial Calibration

A six-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4 Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG

6. Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 120 percent for both sediment and porewater. Based on conversations with the laboratory an additional surrogate, tripentyltin was added and

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QA Review Batch K9805449 (Organotin)

Site. Duwamish River

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historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits	Porewater Limits
Tripropyltin	20 - 195%	20 - 113%
Triphenyltin	20 - 175%	20 - 133%

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following:

Sample	Surrogate	Percent Recovery
98334023	Triphenyltin	123%

Sample results are qualified as estimated (J) when both surrogate recoveries are outside project limits. No qualifiers were assigned as only one surrogate in sample 98334023 was outside the project limits.

7. Laboratory Control Sample (LCS)

LCS recovery goals for tributyltin were established in the project Sampling and Analysis Plan at 60 to 130% for both sediment and porewater. Based on conversations with the laboratory, historical control chart limits of 20 to 138 percent for water and 20 to 164 percent for sediment were also used for data qualification.

All laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980814-LCS	n-Butyltin	20	60-130 (P) 20-164 (L)	98334012 98334017 98334019 98334020 98334022 98334023 98334024



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Sample results for n-Butyltin were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results for n-Butyltin were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for both sediment and porewater.

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
Monbutyltin	10 - 120%

MS/MSD sample percent recoveries and relative percent differences (RPDs) for Tri-n-Butyltin, Di-n-Butyltin and n-Butyltin were not calculated due to high analyte concentrations in the sample. As LCS results were acceptable for Tri-n-butyltin and Di-n-butyltin, samples were not qualified based on matrix spike/matrix spike duplicate results.

9. Field Duplicate Analysis

No field duplicates were associated with this SDG.

10. Sample Analysis

A cursory review of raw data was performed. All results were verified on a second, dissimilar, confirmation GC column. No unusual problems were noted. A duplicate analysis was also performed, RPD results between replicates were less than 35 percent for all analytes.

The case narrative indicated that matrix spike/matrix spike duplicate recoveries for Tri-n-butyltin and Di-n-butyltin for the batch QC sample was not calculated due to high analyte concentrations in the sample. These high analyte levels prevented accurate evaluation of the spike recovery. No other problems were noted.

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11. Laboratory Contact

No laboratory contact was required

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

Data Qualifiers

- U - The compound was analyzed for, but was not detected.
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification
- N - Presumptive evidence of presence of material (tentative identification).

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name 98334012 **Units** ug/Kg (ppb)
Lab Code K9805449-005 **Basis** Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	99	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	57	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	17	J

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

8-31-98

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name 98334017
Lab Code K9805449-010
Test Notes D

Units ug/Kg (ppb)
Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	200	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	48	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	44	J

D

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name 98334019 Units ug/Kg (ppb)
Lab Code K9805449-012 Basis Dry
Test Notes D


Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	110	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	34	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	11	J

D

The MRL is elevated because of matrix interferences and because the sample required diluting

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name	98334020	Units	ug/Kg (ppb)
Lab Code	K9805449-013	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	250	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	45	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/19/98	37	J

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name	98334022	Units	ug/Kg (ppb)
Lab Code	K9805449-015	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	5	5	8/14/98	8/19/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	5	5	8/14/98	8/19/98	170	
Di-n-butyltin	Method	Butyltins-GC	5	5	8/14/98	8/19/98	36	
n-Butyltin	Method	Butyltins-GC	5	5	8/14/98	8/19/98	41	J

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98

Butyltins

Sample Name	98334023	Units	ug/Kg (ppb)
Lab Code	K9805449-016	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	180	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	55	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	ND	10 UJ

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805449
Date Collected: 8/12/98
Date Received: 8/13/98


Butyltins

Sample Name	98334024	Units	ug/Kg (ppb)
Lab Code	K9805449-017	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	ND	
Tri-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	160	
Di-n-butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	49	
n-Butyltin	Method	Butyltins-GC	10	10	8/14/98	8/20/98	26	J

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